

Description

Use of metallocene waxes in powdercoating materials

- 5 The present invention relates to the use of polyolefin waxes synthesized using metallocene catalysts as an additive in powdercoating materials and also to a process for producing pigmented powdercoating materials.

10 The preparation of powdercoating materials by extrusion in co-rotating twin-screw extruders or single-screw kneading apparatus is general knowledge. Powdercoating materials are composed of

- 15 - binders, such as polyester resins, for example, which are crosslinked for example using epoxides, triglycidyl isocyanurate (TGIC), β -hydroxyalkylamine or blocked isocyanates (uretdiones)
- 20 - pigments and fillers, unless the coating materials are clearcoats. The pigments must exhibit appropriately high thermal stability. Examples thereof are phthalocyanines, quinacridones, azo pigments, perylene and perinone pigments, benzimidazolone pigments, anthraquinone pigments, isoindolinone and isoindoline pigments, anthanthrone pigments, dioxazine pigments, quinophthalone pigments and diketopyrrolopyrrole pigments
- 25 - additives, such as devolatilizers, flattening agents, antioxidants, tribological additives, leveling agents, and waxes for enhancing the scratch resistance.

30 In the preparation of powdercoating materials in accordance with the known state of the art the additives used include waxes, in order to achieve the following effects:

- 35 - improving grindability
- preventing deposits in spray equipment and the hose lines thereof
- enhancing scratch resistance
- enhancing abrasion resistance
- improving dispersion harshness of the pigment component
- orienting pigment particles in the case of effect pigments

- improving the color strength
- obtaining flatting effects
- improving the tactility ("soft feel")
- improving lubricity
- 5 - improving metal marking
- improving the ease of incorporation of effect pigments
- influencing rheological properties
- improving blocking resistance
- facilitating devolatilization in the course of extrusion
- 10 - raising the throughput in the course of extrusion

In the preparation of the powdercoating material all of the constituents are first premixed in a mixer, then homogenized in an extruder or kneading apparatus at from 80 to 130°C and finally brought to the ultimate particle size by grinding and classifying. In the preparation of pigmented systems it is particularly important to break down pigment agglomerates into very fine particles and to achieve maximum homogeneity in the dispersion of pigment aggregates, in order to give optimum color strength. At present this is done by introducing mechanical energy via the screw configuration of the extruders or kneading apparatus employed, without using a dispersing assistant.

The wax additives, which are used in a concentration of 0.01-10% by weight, based on the powdercoating material mass, are commonly polyolefin waxes, PTFE waxes, amide waxes, FT paraffins, montan waxes, natural waxes, macrocrystalline and microcrystalline paraffins, sorbitan esters and metallocene waxes.

Suitable polyolefin waxes are primarily polyethylene and polypropylene waxes. They can be prepared by thermal degradation of high-polymer polyolefins or by direct polymerization of corresponding monomers. Suitable polymerization techniques include high-pressure technologies, in which, for example, ethylene is reacted free-radically under high pressures and temperatures to form waxes, and also low-pressure or Ziegler techniques, where the olefin is polymerized using organometallic catalysts at comparatively low pressures and temperatures. The low-pressure technique permits not only the preparation of homopolymer waxes of uniform construction but also the synthesis of copolymers by joint polymerization of two or more olefins.

A variant of the low-pressure technique which has been disclosed more recently is a procedure in which metallocene compounds are used as organometallic catalysts. These metallocene compounds contain titanium, zirconium or hafnium atoms as active species and are generally employed
5 in combination with cocatalysts, examples being organoaluminum compounds or boron compounds, preferably aluminoxane compounds. Polymerization takes place where necessary in the presence of hydrogen as a molar mass regulator. A feature of metallocene processes is that in comparison to the older Ziegler technology it is possible to obtain waxes
10 having a narrower molar mass distribution, more uniform incorporation of comonomer, lower melting points and higher catalyst yields. Polymerization processes of this kind which operate with metallocene catalysts for the preparation of polyolefin waxes are described for example in EP-A-0 571 882.

15 EP-A-0 890 619 describes the use of metallocene polyolefin waxes in printing inks and paints. Not addressed therein are powdercoating materials, with their specific requirements concerning the addition of wax additives.

20 The invention was based on the object of improving the performance properties of powdercoating materials through use of a suitable dispersant.

It has now been found that polyolefin waxes prepared using metallocene
25 catalysts are suitable with particular advantage for use as additives in powdercoating materials. Metallocene polyolefin waxes in particular display improved activity in respect of extrusion properties, flattening effect, lubricity, film hardness, abrasion resistance and dispersion harshness.

30 The invention accordingly provides for the use of polyolefin waxes synthesized using metallocene catalysts as an additive in powdercoating materials.

35 The polyolefin wax is preferably derived from olefins having 3 to 6 carbon atoms or from styrene.

Polyolefin waxes suitable in principle include homopolymers of ethylene and of propylene, copolymers of ethylene and propylene, or copolymers of ethylene or of propylene with one or more 1-olefins. 1-Olefins used are

linear or branched olefins having 3-18 carbon atoms, preferably 3-6 carbon atoms. The 1-olefins may also carry an aromatic substitution.

5 Examples of suitable 1-olefins besides ethylene and propylene are 1-butene, 1-hexene, 1-octene or 1-octadecene, and also styrene. Particular preference is given to homopolymers of ethylene or propylene or to copolymers of ethylene with propylene or 1-butene. In the case of copolymers the ethylene content thereof is preferably 70-99.9%, in particular 80-99% by weight.

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The polyolefin waxes used in accordance with the invention can be prepared either by direct polymerization with metallocene catalysts or by thermal degradation of polyolefin polymers, with the above composition, prepared using metallocene catalysts.

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Used with particular preference are polyolefin waxes having a dropping point of between 70 and 165°C, in particular between 100 and 160°C, a melt viscosity at 140°C (polyethylene waxes) or at 170°C (polypropylene waxes) of between 10 and 10 000 mPas, in particular between 50 and 5000 mPas, and a density at 20°C of between 0.85 and 0.98 g/cm³. Preferred polyolefin waxes have a molecular weight distribution $M_w/M_n < 5$.

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25 Metallocene catalysts for preparing polyolefin waxes or for preparing polyolefin polymers used for thermal degradation are chiral or nonchiral transition metal compounds of the formula M^1L_x . The transition metal compound M^1L_x contains at least one central metal atom M^1 attached to which there is at least one π ligand, e.g., a cyclopentadienyl ligand. Additionally it is possible for substituents, such as halogen, alkyl, alkoxy or aryl groups, to be attached to the central metal atom M^1 . M^1 is preferably 30 an element from main group III, IV, V or VI of the Periodic Table of the Elements, such as Ti, Zr or Hf. By cyclopentadienyl ligand are meant unsubstituted cyclopentadienyl radicals and substituted cyclopentadienyl radicals such as methylcyclopentadienyl, indenyl, 2-methylindenyl, 2-methyl-4-phenylindenyl, tetrahydroindenyl or octahydrofluorenyl radicals. 35 The π ligands can be bridged or unbridged, and both single and multiple bridging – including bridging via ring systems – are possible. The term “metallocene” also embraces compounds containing more than one metallocene fragment, referred to as polynucleometallocenes. These may have any desired substitution pattern and bridging variants. The individual

metallocene fragments of such polynucleometallocenes may be either identical or different from one another (EP-A-0 632 063).

General structure formulae of metallocenes and also their use for preparing polyolefin waxes are given for example in EP-A-0 571 882.

The polyolefin waxes used in accordance with the invention may be employed either as they are or in a polar-modified form. Polar modification can be achieved, for example, by oxidation with air or oxygen-containing gases or by graft attachment of, for example, unsaturated carboxylic acids such as, for instance, maleic acid. Examples of oxidative modification can be found in EP-A-0 890 583, examples of modification with unsaturated carboxylic acids in EP-A-0 941 257.

The polyolefin waxes of the invention can be used both in pure form and also in a blend with further wax components, not prepared using metallocene catalysts, in a fraction of 1-50% by weight. Blending may take place in the melt or by mixing the components in solid form. The following blend components are suitable:

- a) polyethylene glycol
- b) PE waxes,
- c) PTFE waxes,
- d) PP waxes,
- e) amide waxes,
- f) FT paraffins,
- g) montan waxes,
- h) natural waxes,
- i) macrocrystalline and microcrystalline paraffins,
- j) polar polyolefin waxes, or
- k) sorbitan esters
- l) polyamides,
- m) polyolefins,
- n) PTFE,
- o) wetting agents,
- p) silicates.

Additive a) comprises polyethylene glycol, molecular weight range preferably 10 to 50 000 daltons, in particular 20 to 35 000 daltons. The

polyethylene glycol can be admixed in amounts of preferably up to 5% by weight to the composition comprising metallocene wax.

5 Additive b) comprises in preferred embodiments polyethylene homopolymer and copolymer waxes which have not been prepared by metallocene catalysis and which have a number-average molecular weight of from 700 to 10 000 g/mol with a dropping point of between 80 and 140°C.

10 Additive c) comprises in preferred embodiments polytetrafluoroethylene having a molecular weight of between 30 000 and 2 000 000 g/mol, in particular between 100 000 and 1 000 000 g/mol.

15 Additive d) comprises in preferred embodiments polypropylene homopolymer and copolymer waxes which have not been prepared by metallocene catalysis and which have a number-average molecular weight of from 700 to 10 000 g/mol with a dropping point of between 80 and 160°C.

20 Additive e) comprises in preferred embodiments amide waxes preparable by reacting ammonia or ethylenediamine with saturated and/or unsaturated fatty acids. The fatty acids comprise, for example, stearic acid, tallow fatty acid, palmitic acid or erucic acid.

25 Additive f) comprises in preferred embodiments FT paraffins having a number-average molecular weight of from 400 to 800 g/mol with a dropping point of from 80 to 125°C.

30 Additive g) preferably comprises montan waxes, including acid waxes and ester waxes, with a carboxylic acid carbon chain length of C₂₂ to C₃₆.

The ester waxes preferably comprise reaction products of the montanic acids with monohydric or polyhydric alcohols having 2 to 6 carbon atoms, such as ethanediol, butane-1,3-diol or propane-1,2,3-triol, for example.

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Additive h) comprises in one preferred embodiment carnauba wax or candelilla wax.

Additive i) comprises paraffins and microcrystalline waxes which are obtained in the course of petroleum refining. The dropping points of such paraffins are preferably between 45 and 65°C, those of such microcrystalline waxes preferably between 73 and 100°C.

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Additive j) comprises in preferred embodiments polar polyolefin waxes preparable by oxidizing ethylene or propylene homopolymer and copolymer waxes or by grafting them with maleic anhydride. Particular preference is given for this purpose to starting from polyolefin waxes having a dropping point of between 90 and 165°C, in particular between 100 and 160°C, a melt viscosity at 140°C (polyethylene waxes) or at 170°C (polypropylene waxes) of between 10 and 10 000 mPas, in particular between 50 and 5000 mPas, and a density at 20°C of between 0.85 and 0.96 g/cm³.

15 Additive k) comprises in preferred embodiments reaction products of sorbitol with saturated and/or unsaturated fatty acids and/or montan acids. The fatty acids comprise for example stearic acid, tallow fatty acid, palmitic acid or erucic acid.

20 Additive l) comprises preferably ground polyamides, examples being nylon-6, nylon-6,6 or nylon-12. The particle size of the polyamides is preferably in the region of 5-200 µm, in particular 10-100 µm.

25 Additive m) comprises polyolefins, in other words, for example, polypropylene, polyethylene or copolymers of propylene and ethylene of high or low density, with molar weights of preferably from 10 000 to 1 000 000 D, in particular from 15 000 to 500 000 D, as the numerical average of the molecular weight, whose particle size as a result of grinding is in the region of preferably 5-200 µm, in particular 10-100 µm.

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Additive n) comprises thermoplastic PTFE having a molar weight of preferably 500 000-10 000 000 D, in particular 500 000-2 000 000 D, as numerical average, whose particle size as a result of grinding is in the region of preferably 5-200 µm, in particular 10-100 µm.

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Additive o) comprises amphiphilic compounds which generally lower the surface tension of liquids. The wetting agents comprise, for example, alkyl ethoxylates, fatty alcohol ethoxylates, alkylbenzenesulfonates or betaines.

Additive p) comprises silicates which are not used as a filler or pigment in the formulas. Preference is given to using silicas or talc.

- 5 The mixing ratio of ingredient a) to ingredients b) to p) can be varied in the range from 1 to 50% by weight of a) to from 1 to 50% by weight of b) to p). If a mixture of two or more of ingredients b) to p) is used then the amount specified is valid for the sum of the amounts of those ingredients.

- 10 In one preferred embodiment the waxes are used in micronized form for the purpose according to the invention. Particular preference is given to using polyolefin wax and, if desired, admixed auxiliaries and additives in the form of an ultrafine powder having a particle size distribution $d_{90} < 40 \mu\text{m}$.

- 15 The invention further provides a process for preparing powdercoating materials from binders, pigments and fillers and also customary auxiliaries, which comprises adding an additive as per the present invention.

Examples

20 Table 1: Raw materials

Product	Acid number	Dropping point	Viscosity
metallocene PE wax 1)	0 mg KOH/g	124°C	250 mPas (140°C)
metallocene PP wax 2)	0 mg KOH/g	135°C	40 mPas (170°C)
oxidized metallocene PE wax 3)	20 mg KOH/g	114°C	200 mPas (120°C)
Ziegler PE wax	0 mg KOH/g	125°C	300 mPas (140°C)
Ziegler PP wax	0 mg KOH/g	160°C*	1500 mPas (170°C)
oxidized PE wax	20 mg KOH/g	114°C	200 mPas (120°C)
amide wax	6 mg KOH/g	140°C	10 mPas (150°C)
montan wax 1	17 mg KOH/g	82°C	30 mPas (100°C)
montan wax 2	14 mg KOH/g	100°C	300 mPas (120°C)
PTFE wax	-	-	-
carnauba wax	9 mg KOH/g	82°C	30 mPas (90°C)
FT paraffin	0 mg KOH/g	110°C	15 mPas (120°C)

1) Preparation in accordance with EP-A-0 571 882

2) Preparation in accordance with EP-A-0 890 584

3) Preparation in accordance with EP-A-0 890 583

*) Ring & ball softening point

Table 2: Specimens tested

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M = inventive example

V = comparative example

All specimens were micronized to a DV₅₀ of approximately 8 μ m.

Code	Wax 1	Wax 2	Wax 3	Mixing ratio
M1	oxidized metallocene PE wax	carnauba wax		1:1
V1	oxidized PE wax	carnauba wax		1:1
M2	metallocene PE wax	oxidized metallocene PE wax		7:3
V2	PE wax	oxidized PE wax		7:3
M3	metallocene PE wax	amide wax		1:1
V3	PE wax	amide wax		1:1
M4	metallocene PE wax	PTFE wax		9:1
V4	PE wax	PTFE wax		9:1
M5	metallocene PE wax	oxidized metallocene PE wax	PTFE wax	12:7:1
V5	PE wax	oxidized PE wax	PTFE wax	12:7:1
M6	metallocene PP wax	amide wax		1:1
V6	PP wax	amide wax		1:1
M7	metallocene PP wax	amide wax		5:1
V7	PP wax	amide wax		5:1
M8	metallocene PP wax	metallocene PE wax		1:1
V8	PP wax	PE wax		1:1
M9	metallocene PP wax	oxidized metallocene PE wax		1:1
V9	PP wax	oxidized PE wax		1:1
M10	oxidized metallocene PE wax	montan wax 1	montan wax 2	2:1:1
V10	oxidized PE wax	montan wax 1	montan wax 2	2:1:1
M11	metallocene PE wax	oxidized metallocene PE wax	sorbitan tristearate	1:1:1
V11	PE wax	oxidized PE wax	sorbitan tristearate	1:1:1
M12	metallocene PE wax	FT paraffin		5:1
V12	PE wax	FT paraffin		5:1

Table 3: Incorporation of micronized waxes into a blue hybrid powdercoating material for the purpose of flattening

The waxes were mixed with the individual raw materials with one another in a high-speed mixer and then the dry mixture was extruded on a laboratory twin-screw extruder (PC19-25 from APV) at 110°C and 250 rpm, ground to < 125 µm and applied to aluminum or steel panels by means of a spraygun with corona charging. After baking (15 minutes at 180°C) the coated panels were stored in a controlled-climate chamber for 24 hours, after which the gloss (60°) was measured.

	Wax	Wax fraction	Gloss (60° angle)
Example 1	no wax	0%	96
Example 2	M2	0.5%	90
Example 3	V2	0.5%	92
Example 4	M2	1.0%	88
Example 5	V2	1.0%	90
Example 6	M2	2.0%	80
Example 7	V2	2.0%	83
Example 8	M3	0.5%	93
Example 9	V3	0.5%	96
Example 10	M3	1.0%	90
Example 11	V3	1.0%	94
Example 12	M3	2.0%	88
Example 13	V3	2.0%	91
Example 14	M4	0.5%	30
Example 15	V4	0.5%	35
Example 16	M4	1.0%	7
Example 17	V4	1.0%	10
Example 18	M4	2.0%	7
Example 19	V4	2.0%	8
Example 20	M6	0.5%	90
Example 21	V6	0.5%	94
Example 22	M6	1.0%	85
Example 23	V6	1.0%	90
Example 24	M6	2.0%	75
Example 25	V6	2.0%	78
Example 26	M10	0.5%	93

Example 27	V10	0.5%	96
Example 28	M10	1.0%	88
Example 29	V10	1.0%	92
Example 30	M10	2.0%	81
Example 31	V10	2.0%	87
Example 32	M12	0.5%	92
Example 33	V12	0.5%	95
Example 34	M12	1.0%	87
Example 35	V12	1.0%	92
Example 36	M12	2.0%	82
Example 37	V12	2.0%	87

In all of examples 2 to 37 the inventive specimens (M1-M12) exhibit better results in each case than the comparatives (V1-V12).

- 5 Table 4: Incorporation of micronized waxes into a blue hybrid powdercoating material for the purpose of sliding friction

10 The waxes were mixed with the individual raw materials with one another in a high-speed mixer and then the dry mixture was extruded on a laboratory twin-screw extruder (PC19-25 from APV) at 110°C and 250 rpm, ground to < 125 µm and applied to aluminum or steel panels by means of a spraygun with corona charging. After baking (15 minutes at 180°C) the coated panels were stored in a controlled-climate chamber for 24 hours, after which the sliding friction (according to Altek) was measured.

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	Wax	Wax fraction	Sliding friction
Example 38	no wax	0%	0.33
Example 39	M3	0.5%	0.27
Example 40	V3	0.5%	0.29
Example 41	M3	1.0%	0.25
Example 42	V3	1.0%	0.27
Example 43	M3	2.0%	0.21
Example 44	V3	2.0%	0.25
Example 45	M4	0.5%	0.22
Example 46	V4	0.5%	0.24
Example 47	M4	1.0%	0.20
Example 48	V4	1.0%	0.23

Example 49	M4	2.0%	0.15
Example 50	V4	2.0%	0.18
Example 51	M11	0.5%	0.17
Example 52	V11	0.5%	0.19
Example 53	M11	1.0%	0.16
Example 54	V11	1.0%	0.17
Example 55	M11	2.0%	0.13
Example 56	V11	2.0%	0.15

In all of examples 38 to 56 the inventive specimens (M3, M4, M11) exhibit better results in each case (lower sliding friction) than the comparatives (V3, V4, V11).

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Table 5: Incorporation into a white hybrid powdercoating material for the purpose of improving the pencil hardness

The waxes were mixed with the individual raw materials with one another in a high-speed mixer and then the dry mixture was extruded on a laboratory twin-screw extruder (PC19-25 from APV) at 110°C and 250 rpm, ground to < 125 μ m and applied to aluminum or steel panels by means of a spray gun with corona charging. After baking (15 minutes at 180°C) the coated panels were stored in a controlled-climate chamber for 24 hours, after which the pencil hardness (according to Wolff-Wilborn) was determined.

	Wax, in each case 1% based on overall formula	Wolff-Wilborn pencil hardness
Example 57	no wax	2B
Example 58	M2	HB
Example 59	V2	B
Example 60	M3	F
Example 61	V3	HB
Example 62	M4	F
Example 63	V4	HB
Example 64	M6	F
Example 65	V6	B
Example 66	M7	H
Example 67	V7	B
Example 68	M10	HB
Example 69	V10	HB

In all of examples 57 to 69 it was possible to achieve a higher pencil hardness in each case with the inventive specimens than with the comparatives.

- 5 Table 6: Incorporation into a white hybrid powdercoating material for the purpose of improving the abrasion resistance

10 The waxes were mixed with the individual raw materials with one another in a high-speed mixer and then the dry mixture was extruded on a laboratory twin-screw extruder (PC19-25 from APV) at 110°C and 250 rpm, ground to < 125 μ m and applied to aluminum or steel panels by means of a spray gun with corona charging. After baking (15 minutes at 180°C) the coated panels were stored in a controlled-climate chamber for 24 hours, after which the abrasion test on the Taber Abraser was determined.

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	Wax, in each case 1% based on overall formula	Abrasion test after 200 revolutions
Example 70	no wax	52 mg
Example 71	M2	48 mg
Example 72	V2	50 mg
Example 73	M3	35 mg
Example 74	V3	41 mg
Example 75	M4	25 mg
Example 76	V4	42 mg
Example 77	M6	20 mg
Example 78	V6	46 mg
Example 79	M7	15 mg
Example 80	V7	38 mg
Example 81	M10	25 mg
Example 82	V10	40 mg

In all of examples 70 to 82 the inventive specimens exhibit better results (lower abrasion) in each case than the comparatives.

- 20 Table 7: Incorporation into a blue hybrid powdercoating material for the purpose of improving the dispersion harshness of pigments

The waxes were mixed with the individual raw materials with one another in a high-speed mixer and then the mixture was extruded on a laboratory twin-screw extruder (PC19-25 from APV) at 110°C and 250 rpm, in the course of which it was necessary to adjust the metering amount for a power consumption of 70% on the extruder, after which in this case the throughput was ascertained, and then the mixture was ground to < 125 µm and applied to aluminum or steel panels by means of a spraygun with corona charging. After baking (15 minutes at 180°C) the coated panels were stored in a controlled-climate chamber for 24 hours and thereafter the dispersion harshness was determined via the depth of color.

	Wax, in each case 1% based on overall formula	Depth of color
Example 83	no wax	100%
Example 84	M1	110%
Example 85	V1	107%
Example 86	M2	105%
Example 87	V2	100%
Example 88	M3	107%
Example 89	V3	102%
Example 90	M6	105%
Example 91	V6	100%
Example 92	M8	107%
Example 93	V8	105%
Example 94	M10	120%
Example 95	V10	112%

In all of examples 83 to 95 the inventive specimens exhibit better results (greater dispersion harshness) in each case than the comparatives.

Table 8: Incorporation into a blue hybrid powdercoating material for the purpose of improving the throughput

The waxes were mixed with the individual raw materials with one another in a high-speed mixer and then the mixture was extruded on a laboratory twin-screw extruder (PC19-25 from APV) at 110°C, in the course of which it was necessary to adjust the metering amount for a power consumption of

60% at 250 rpm on the extruder, after which in this case the throughput was ascertained.

	Wax	Wax fraction	Throughput per minute
Example 96	no wax	0%	7500 g
Example 97	M2	0.5%	9000 g
Example 98	V2	0.5%	8600 g
Example 99	M2	1.0%	9200 g
Example 100	V2	1.0%	8700 g
Example 101	M2	2.0%	9500 g
Example 102	V2	2.0%	9000 g
Example 103	M4	0.5%	8500 g
Example 104	V4	0.5%	8200 g
Example 105	M4	1.0%	8700 g
Example 106	V4	1.0%	8400 g
Example 107	M4	2.0%	8800 g
Example 108	V4	2.0%	8400 g
Example 109	M10	0.5%	8000 g
Example 110	V10	0.5%	7500 g
Example 111	M10	1.0%	10 100 g
Example 112	V10	1.0%	9500 g
Example 113	M10	2.0%	11 000 g
Example 114	V10	2.0%	10 700 g
Example 115	M12	0.5%	7600 g
Example 116	V12	0.5%	7500 g
Example 117	M12	1.0%	8500 g
Example 118	V12	1.0%	8500 g
Example 119	M12	2.0%	9000 g
Example 120	V12	2.0%	8500 g

- 5 In all of examples 96 to 120 the inventive specimens exhibit better results in each case (higher throughput) than the comparatives.

Table 9: Incorporation into a blue hybrid powdercoating material for the purpose of improving the power consumption

The waxes were mixed with the individual raw materials with one another in a high-speed mixer and then the mixture was extruded on a laboratory twin-screw extruder (PC19-25 from APV) at 110°C, where the metering was adjusted to 3.0 and subsequently the power consumption was measured.

	Wax	Wax fraction	Power consumption
Example 121	no wax	0%	69%
Example 122	M2	0.5%	61%
Example 123	V2	0.5%	62%
Example 124	M2	1.0%	60%
Example 125	V2	1.0%	62%
Example 126	M2	2.0%	57%
Example 127	V2	2.0%	61%
Example 128	M4	0.5%	65%
Example 129	V4	0.5%	67%
Example 130	M4	1.0%	62%
Example 131	V4	1.0%	64%
Example 132	M4	2.0%	59%
Example 133	V4	2.0%	62%
Example 134	M10	0.5%	59%
Example 135	V10	0.5%	63%
Example 136	M10	1.0%	54%
Example 137	V10	1.0%	60%
Example 138	M10	2.0%	51%
Example 139	V10	2.0%	54%
Example 140	M11	0.5%	60%
Example 141	V11	0.5%	63%
Example 142	M11	1.0%	56%
Example 143	V11	1.0%	61%
Example 144	M11	2.0%	54%
Example 145	V11	2.0%	57%
Example 146	M12	0.5%	60%
Example 147	V12	0.5%	66%
Example 148	M12	1.0%	59%
Example 149	V12	1.0%	63%
Example 150	M12	2.0%	58%
Example 151	V12	2.0%	60%